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(54) Title: DENTAL PROPHYLAXIS COMPOSITIONS AND THEIR USE

(57) Abstract

The application discloses new dental prophylactic cleaning and polishing agents comprising magnesium silicate particles obtained by thermally treating naturally-occurring or synthetic magnesium silicates.

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Background of Invention

Dental Prophylaxis Compositions and Their Use

Field of the Invention

The present invention relates to the field of dental prophylaxis and more specifically to prophylaxis paste cleaning and polishing compositions.

These compositions have the ability to impart a smooth, highly polished surface to tooth enamel and to effectively remove all types of exogenous stains and accumulations from the teeth without resulting in undue abrasion of the enamel, dentin, or cementum. Reaccumulations of dental plaque and pellicle and occurrence and reformation of dental calculus on oral hard tissue are markedly reduced, thereby, significantly reducing the incidence of gingivitis and periodontal disease. Additionally, as a means of contributing to the partial control of dental caries, effective fluoride anticariogenic adjuvants, such as sodium fluoride, stannous fluoride, sodium monofluorophosphate, or acidulated phosphate fluoride, may be incorporated in such compositons. Other anticariogenic agents, such as aluminum carboxylates (U. S. Patent Nos. 4,042,680 and 4,108,981) may also be advantageously employed in such compositions.

Description of the Prior Art

In the past, prophylaxis pastes have been used for the removal of exogeneous stains that could not be removed by the routine use of a dentifrice and toothbrush. Even at the present, many widely used dental prophylaxis products contain pumice, silica, or other hard materials of a relatively large particle size in order to achieve fast and thorough cleaning. It is apparent that excessive abrasion and scratching of the enamel increase the rate of

reformation of exogeneous stains and produce a low degree of enamel polish. It has been shown in several studies that a smooth, highly polished tooth surface is less receptive to reformation and retention of plaque, exogenous debris, pellicle, stains, and dental calculus.

Although significant improvements in the polishing efficacy of prophylaxis compositions have been achieved in recent years through the use of zirconium silicate (U. S. Patents Nos. 3,330,732 and 3,378,445), alumina (U. S. Patent No. 3,670,076), and feldspar (U. S. Patent No. 3,892,843), these agents still fall short of imparting maximum levels of polish during a typical, short prophylaxis treatment. Additionally, in order to achieve good cleaning properties with these materials, large particles are necessary; however, such particle sizes cause undesirably high abrasiveness and do not polish well. To obtain polishing, especially with alumina or zirconium silicate, it is necessary to include small-sized particles, which have little cleaning effect.

The beneficial effects, in terms of a reduction in the incidence of dental caries, resulting from the incorporation of water-soluble fluoride salts, such as sodium fluoride, stannous fluoride, sodium monofluorophosphate, or acidulated phosphate fluoride, are well known. However, efforts to utilize such salts in prophylaxis paste compositions have been handicapped by the tendency of fluoride and/or tin(II) ions to be inactivated and rendered unavailable by other ingredients, particularly the abrasive component of such compositions. In general, while prophylactic abrasives in therapeutic products used today are to varying degrees compatible with fluoride agents, there is a wide variation in compatibility. Abrasives containing polyvalent cations such as calcium and iron, either in their crystal structure or as impurities, are usually not particularly compatible.

Thus, prior art materials intended for use as cleaning and polishing constituents of prophylactic compositions

have been unsatisfactory in one or more of the following respects, namely poor cleaning and polishing performances (especially with respect to inhibition of reaccumulation of dental calculus, pellicle, plaque, and exogenous stains), incompatibility with fluoride-containing anticariogenic agents, and adverse scratching and abrasion.

Summary of Invention

In accordance with the present invention, it has been found that new and more effective dental prophylactic preparations may be obtained by incorporating therein, as cleaning and polishing constituents, naturally occurring or synthetic magnesium silicate particles calcined at a temperature in the range of about 800° to 1200°C. Preferably, the magnesium silicate particles are hydrous in nature. Substantially none of the calcined particles is retained on an 80 mesh screen (177 microns) and essentially all pass through a 100 mesh screen (149 microns). Desirably, all of the particles are less than about 150 microns in diameter, and advantageously the median particle size lies in the range of 10-40 microns. Preferably, the calcined magnesium silicate particles are predominantly of the magnesium metasilicate form.

It has been discovered that magnesium silicates, when calcined at the desired temperature ranges and when prepared in the desired particle size ranges, can be used as a combined cleaning and polishing agent, overcoming the aforementioned problems of the prior art. Tests show that excellent cleaning and polishing results are obtained from a range of particle sizes within given limits, so that all particles participate in both functions, regardless of size. Cleaning and polishing are achieved with less scratching of the enamel surface that with prior materials. Also, it has been found that larger particles of calcined magnesium silicate, in contrast to other dental prophylactic abrasives, are less abrasive than smaller

particles of other materials, which accentuate the advantage.

It has further been found that the novel cleaning and polishing agents of the present invention may be used with non-toxic amounts of water-soluble anticariogenic adjuvants, such as sodium fluoride, stannous fluride, sodium monofluorophosphate, or acidulated phosphate fluoride. In addition, with calcined magnesium silicates, the amount of anticariogenic agent added can be substantially reduced and yet superior reduction of enamel solubility is obtained. Such property has, among others, the advantage that taste problems are minimized or avoided.

It has likewise been discovered that application of prophylactic preparations of the present invention to the teeth provides a novel method for cleaning and polishing teeth and for reducing the incidence of gingivitis.

Through the use of the cleaning and polishing agents of the present invention, the difficulties experienced with prior art dental prophylaxis cleaning and polishing agents may be overcome, and compositions of the present invention may therefore be used to formulate prophylaxis pastes with superior cleaning and polishing capabilities and with enhanced anticariogenic ion compatibilities.

Description of Preferred Embodiments

In accordance with the subject invention, it has been found that optimal cleaning and polishing characteristics for a prophylaxis paste cleaning and polishing agent are exhibited by calcined, naturally-occurring or synthetic magnesium silicate particles. The magnesium silicate particles are ordinarily hydrated prior to calcining, and after calcining are predominantly of the magnesium metasilicate form. In addition, it has been found that the cleaning and polishing compositions of the present invention may be advantageously used with water-soluble fluoride ion-containing anticariogenic adjuvants, such as

sodium fluoride, stannous fluoride, sodium monofluorophosphate, or acidulated phosphate fluoride.

As a result of the foregoing, the calcined magnesium silicates of the character described find utility in therapeutic dental prophylaxis compositions (i.e. compositions containing at least one anticariogenic ionic adjuvant in combination with a compatible cleaning and polishing agent and designed to reduce the incidence and severity of dental caries) or in dental prophylaxis compositions which, although not containing fluorides or other anticariogenic agents, nonetheless have therapeutic utility in reducing gingival disease.

The calcined magnesium silicates of this invention may be obtained by calcining (i.e. heat treating) a variety of naturally-occurring magnesium silicate minerals (which have been mined, cleaned, dried and fractionated) and synthetic magnesium silicate compounds.

Naturally-Occurring Magnesium Silicate Minerals

The naturally-occurring minerals suitable for this thermal transformation are composed of several representatives of the phyllosilicate group of minerals. These are all structurally similar in that they are composed of superimposed layers of two-dimensional silica tetrahedra and two-dimensional octahedra consisting of two sheets of closely packed oxygens or hydroxyls in which aluminum, iron, or magnesium atoms are embedded in octrahedral coordination. However, only the magnesium-rich members of the phyllosilicate group exhibit the unique properties of the subject invention. Among these are clay minerals, such as smectite (e.g. hectorite $[(Mg_{3-x}Li_x)Si_4O_{10}(OH)_2]$, montmorillonite or bentonite $[(Al_{2-x}Mg_x)Si_4O_{10}(OH)_2]$, and saponite $[(Mg_{3-x}Al_x)(Si_{4-y}Al_y)O_{10}(OH)_2)]$; sepiolite (meerschaum $[Mg_2Si_3O_8 2H_2O]$); chlorite $[(Mg,Al,Fe)_6\ (Si,Al)_4O_{10}(OH)_8]$; and mica-type minerals (e.g. talc $[Mg_3Si_4O_{10}(OH)_2]$ and phlogopite $([KMG_3AlSi_3O_{10}(OH)_2])$.

Synthetic Magnesium Silicates

The synthetic hydrous magnesium silicates which are suitable consist of colloidal magnesium silicate (synethtic smectites similar in composition to saponite and hectorite), magnesium trisilicate ($Mg_2Si_3O_8$), and amorphous hydrous magnesium silicate (empirical formula, $Mg_2Si_5O_{12}$). One preferred material is an amorphous hydrous magnesium silicate available under the trademark "BRITESORB".

As used herein the term magnesium silicate should unless otherwise qualified be understood to refer to naturally occurring as well as synthetic magnesium silicates as described here.

While magnesium silicates containing some iron in their structures may be used for cleaning and polishing the teeth, the materials should be low in iron ions when combined with fluoride adjuvants for therapeutic treatment. Similarly, calcium-containing impurities such as dolomite and calcite, which occur frequently in depositis of talcs and clay minerals, must be removed from the magnesium silicate materials prior to calcination if a fluoride agent is to be added. Several naturally-occurring minerals which have been found to be particularly useful have approximate compositions by weight as in Table 1.

TABLE 1

Chemical Compositions of Naturally-Occurring Hydrous Magnesium Silicates

Component	Per Cent				
	Chlorite	Hectorite	Saponite	Sepiolite	Steatite
SiO_2	30.5	58.5	61.1	56.0	61.3
Al_2O_3	22.4	0.2	9.3	4.0	0.9
Fe_2O_3	2.1	0.2	0.9	1.0	0.9
MgO	31.1	26.8	13.7	20.0	30.3
CaO	0.15	1.1	2.7	0.5	0.09
Na_2O	0.08	2.6	2.9	1.4	0.07
K_2O	0.20	0.1	0.3	1.4	0.01
Li_2O	---	1.2	---	---	---
TiO_2	0.08	---	0.1	---	0.05

In their natural state all the described clay and mica-type minerals are of little value as abrasive agents because of their softness, their colloid formation, swelling, gellation in water, and their reactivity, adsorptivity, and ion exchangeability. However, thermal treatment of these minerals causes significant changes in their structures. At lower temperatures (usually 700°C. or lower) dehydration of most clay minerals is reversible because only adsorbed or interlayer water is lost and no structural changes occur. Heat treatment at higher temperatures (i.e. calcination) causes the minerals to undergo structural changes since hydroxyl water of the crystal lattice is lost and entirely new materials with totally different properties are produced. The types of alterations and the temperatures at which they occur vary with the chemical composition, structure and particle size of the mineral.

In accordance with this invention the magnesium silicates are calcined at a temperature lying in the range

of about 800°C to 1200°C. Calcining may be achieved by heating in saggers in a furnace or by means of a rotary kiln wherein the degree of calcination may be controlled by altering the feed rate of material to the calciner, by varying the calcination residence time or the thickness of the material bed in the calciner, or by other methods known in the art. If the temperature does not reach 800°-900°C., the magnesium silicates remain in an incompletely dehydrated form, a material which is insufficiently hard to clean and polish satisfactorily from a dental standpoint. Material which has been calcined in the range of 800° to 1000°C. is predominantly magnesium metasilicate in the form of enstatite-type crystals. However, if more highly calcined (i.e. is subjected to temperatures of up to about 1200°C.), materials such as cristobalite (SiO_2) are formed. The amount of cristobalite formed is dependent on the ratio of magnesium to silicon in the starting material. The higher the magnesium content, the more favored is the production of the preferred metasilicate form. Magnesium metasilicate (enstatite) is the preferred reaction product because of its lower hardness and unique crystal structure. Material containing large amounts of cristobalite is unsatisfactory from a dental standpoint because of its tendency to abrade the tooth enamel unless reduced in size by milling or grinding. As a consquence, the calcined hydrous magnesium silicates of this invention are predominantly of the magnesium metasilicate (enstatite) form.

Smectites, because of the many possible substitutions and interlayer cations, show wide variations of temperature for the loss of hydroxyl lattice water. For montmorillonites with low to moderate substitutions of iron and magnesium for aluminum, rapid loss of hydroxyl water begins at about 5000°C., and the dehydration is practically complete at 800°C. Some smectites which have a moderately high magnesium content show a lowering of the dehydration temperature. On the other hand, the magnesium-rich

hectorite, in which the hydroxyls are partially replaced by fluorine, dehydrates at a higher temperature than the aluminous smectites. The structure of many montmorillonites persists to temperatures of the order of 800° to 900°C. when phase changes of the crystal lattice result.

The products formed as a result of high temperature phase changes are entirely dependent on the type and extent of substitution and are often complex. Montmorillonite seems to develop quartz if there is substantially no substitution in the tetrahedral sheet and spinel if there is substantial substitution of aluminum in tetrahedral positions. Since most substitutions occur in the octahedral sheet, the composition of that sheet often determines the outcome; nevertheless, the presence of exchangeable interlayer cations also can have a pronounced effect on the formation of crystal phases. Typically, low-iron montmorillonites develop enstatite ($MgSiO_3$), mullite ($Al_6Si_2O_{13}$), quartz (SiO_2), crystobalite (SiO_2), and sometimes forsterite (Mg_2SiO_4) at about 1000°C. depending on the amount of magnesium present and also on the amount of exchangeable cations.

The identification of such products is usually accomplished by x-ray diffraction, but the patterns are often rather diffuse and are not always identifiable. The exact crystalline material resulting from the calcination step is not critical since it is the material's behavior, not its exact chemical composition or precise crystal structure, that is critical.

Pure talc is heat-stable up to 900°C. Chemically combined water of the talc mineral is lost in the region of 900° to 1000°C., and formation of enstatite crystals begin to occur.

As noted, the starting magnesium silicate minerals are ordinarily hydrous in nature. However, is is possible to employ dehydrated magnesium silicates in accordance with this invention since such materials are also transformed

into the desired metasilicate form upon calcination regardless of the initial extent of hydration.

After calcining in the temperature range of 1000°C. to 1200°C., many of the materials agglomerate into large masses and grinding and/or milling are required to obtain an abrasive having a particle size distribution lying in the range found to be useful in dental prophylaxis compositions. Economical dry grinding processes such as conentional ball-milling may be employed, followed by screening through standard mesh sieves to separate incompletely degraded agglomerates. The preparation of suitably sized particles of the calcined magnesium silicates may also be accomplished by other conventional techniques well known to the art.

The calcined magnesium silicates of the present invention have a particle size range such that substantially none of the particles is retained on an 80 mesh screen (177 microns) and the size range is essentially less than 100 mesh (149 microns). Preferably, the particle size distribution falls into the ranges as shown in Table 2 which were determined by means of a Micromerograph.

TABLE 2

Particle Size Distribution Range of Calcined Magnesium Silicates

Particle Size	Weight (%)
70	0-10
60; 70	0-10
50; 60	0-15
40; 50	2-20
30; 40	5-25
20; 30	5-25
10; 20	10-40
5; 10	5-30
5	0-20
Median	x=10-40 microns

Cleaning and polishing without serious scratching are pronounced when the particle size range is such that a majority of the particles by weight pass through a 200 mesh screen. Within the indicated size range, the particles are effective for both cleaning and polishing. Hence, it is unnecessary to be meticulous in proportioning large and small particles so as to obtain a blend that can both clean and polish teeth. The value of this can be better understood by recognizing that for prophylactic cleaning and polishing of children's teeth, very little actual scouring is required, but polishing is important because immature teeth have alow luster. In contrast, whereas for adult's teeth, which are often stained by tobacco, coffee, tea, etc., substantial cleaning ability is required. With prior art cleaning and polishing agents, separate mixes of finer particle sizes are made to avoid excess and unnecessary scratching and abrasion of the teeth, and other mixes of larger sized particles are made to perform the difficult cleaning adequately.

With the calcined magnesium silicates of the present invention, it is possible to use a single overall size range of particles for both children's and adult's teeth. Moreover, in both situations the degree of abrasivity or scratching to the oral hard tissues is less than with prior art materials. Thus, this product is considerably safer to use than prior art materials, because it cleans and

polishes as well or better than prior art materials with less deleterious abrasion and scratching.

The calcined magnesium silicate cleaning and polishing agents of the present invention may be applied directly to the teeth as a powder in aqueous slurry form. However, it is preferred that the agent be applied in the form of a prophylaxis paste composition. The cleaning and polishing agent is provided in the paste composition within a range of about 30-95% by weight of the overall composition, depending upon the particular formulation desired, as is well known to one skilled in the art. Where desired, a portion of the calcined magnesium silicates of this invention or combinations thereof may be replaced by compatible fillers, extenders, or other abrasives such as uncalcined talcs, uncalcined magnesium silicates, aluminum silicates, alumina, zirconium silicate, insoluble sodium metaphosphate, silicas, etc., and mixtures of these other agents as well as other dental abrasive materials. The prophylaxis paste may be prepared in a conventional manner and usually includes additional ingredients that render the overall composition commercially acceptable. For example, prophylaxis pastes typically contain conventional components such as water, binders, humectants, flavoring agents, sweeteners, detergents, and the like, in the range of up to approimately 50% by weight. Through the use of a prophylaxis paste of the character described, it is possible to obtain clean, yet highly polished, oral hard tissues during the infrequent (i.e, semi-annual) professional prophylaxis treatments performed by a dentist or dental hygienist.

Furthermore, it is preferred that anticariogenic agents be incorporated in such prophylaxis pastes so that the advantages of such agents may be obtained in addition to the cleaning and polishing advantages of the abrasive component. The anticariogenic agent may comprise one or more water-soluble fluoride salts, including NaF, Snf_2, Na_2PO_3F as well as acidulated phosphate fluoride (APF)

mixtures. Other suitable fluoride adjuvants include KF, LiF, SnF_4, InF_3, PbF_2, FeF_2, TiF_4, and NH_4F, as well as more complex water-soluble fluoride-containing adjuvants such as fluorosilicates, e.g. Na_2SiF_6; fluorozirconates, e.g. $CaZrF_6$, Na_2ZrF_6, K_2ZrF_6, $SnZrF_6$, $InZrF_7$; fluorostanites, e.g. $NaSnF_3$, $KSnF_3$, $NaSn_2F_5$; fluoroborates, e.g. $NaBF_4$; fluorotitanates, e.g. $NaTiF_5$; fluorogermanates, e.g. K_2GeF_6, $Zr(GeF_6)_2$, $ZrOGeF_6$, $In_2(GeF_6)_3$; and mixed halides, e.g. SnClF and Sn_2ClF_3.

When used in combination with a fluoride-containing anticariogenic adjuvant in aqueous solution, the tendency of the calcined magnesium silicates of the present invention to react with or deactivate the fluoride adjuvant is substantially nil. Because of the compatibility of these cleaning and polishing agents with fluoride compounds throughout the biologically feasible pH range, the anticariogenic effect of the fluoride can be obtained using lower concentrations of the adjuvant than heretofore possible with prior art dental prophylactic abrasives. This eliminates or at least reduces bad flavor problems and the nauseating effects of conventional larger quantities of fluoride-containing adjuvants, especially stannous fluorides.

Fluoride-containing adjuvants are employed in pastes of the invention at a non-toxic concentration sufficient to significantly reduce the incidence of dental caries in patients. This concentration may range widely, and depends, at least in part, upon the nature of the chosen adjuvant. In general, satisfactory results may be obtained within the range of about 0.1 to 20% by weight of the paste composition (calculated as fluoride ion). When NaF, Na_2PO_3F, or HF are utilized, such compounds are preferably employed at levels of about 0.5 to 5% by weight of the paste. With SnF_2 a concentration range of approximately 1 to 10% is acceptable.

Advantageously, a source of stable phosphate (e.g. phosphoric acid or sodium orthophosphates) may be used in

conjunction with the fluoride agent in order to enhance its anticariogenic activity.

In addition, other suitable anticariogenic adjuvants, such as the aluminum carboxylate complexes described in U. S. Patents Nos. 4,042,680 and 4,108,981 and other non-toxic water soluble sources of aluminum ions may also be employed to produce anticariogenic prophylaxis paste compositions in accordance with the subject invention.

Compositions of exemplary prophylaxis paste preparations employing the cleaning and polishing agents of the present invention are given in the following examples:

EXAMPLE 1

Constituent	% by Weight
Sepiolite (Calcined at 1100°C.)	45.0
Distilled Water	20.9
Glycerin	15.0
Sorbitol (70% aqueous solution)	16.0
Veegum (Magnesium aluminum silicate)	0.5
Sodium Carboxymethyl Cellulose	1.0
Sodium Saccharin	0.5
Flavor	1.0
Methyl p-hydroxybenzoate	0.1
	100.0%

EXAMPLE 2

Constituent	% by Weight
Steatite Talc (calcined at 1200°C.)	94.5
Propylene Glycol	4.5
Hydroxyethyl Cellulose	0.2
Sodium Saccharin	0.3
Flavor	0.5
	100.0%

The above composition may be mixed to any consistency desired with water or fluoride solution immediately before use according to the preferences of the dentist or dental hygienist.

EXAMPLE 3

Constituent	% by Weight
Synthetic Amorphous Hydrous Magnesium Silicate (calcined at 1100°C.)	44.0
Distilled Water	24.4
Propylene Glycol	13.0
Sorbitol (70% aqueous solution)	13.0
Laponite (Synthetic colloidal magnesium silicate)	0.8
Sodium Carboxymethyl Cellulose	0.7
Sodium Saccharin	0.5
Flavor	1.0
Sodium Lauryl Sulfate	0.5
Methyl p-hydroxybenzoate	0.1
Sodium Fluoride, NaF	2.0
	100.0%

EXAMPLE 4

Constituent	% by Weight
Hectorite (calcined at 900°C.)	52.1
Distilled Water	18.0
Propylene Glycol	20.0
Hydroxyethyl Cellulose	0.9
Sodium Saccharin	1.0
Flavor	2.0
Trisodium Citrate	1.0
Stannous Fluoride, SnF_2	5.0
	100.0%

EXAMPLE 5

Constituent	% by Weight
Magnesium Trisilicate (calcined at 1000°C.)	51.0
Distilled Water	19.5
Glycerin	4.7
Propylene Glycol	12.5
Sorbitol (70% aqueous solution)	2.7
Bentone (Organo-clay gellant)	1.5
Sodium Saccharin	0.4
Flavor	0.6
Sodium Fluoride, NaF	4.0
Sodium Dihydrogen Phosphate, NaH_2PO_4	2.3
Miscellaneous	0.8
	100.0%

Experimental Evaluations

The superiority of the calcined magnesium silicate prophylaxis cleaning and polishing agents disclosed herein as compared with other abrasives has been substantiated by the following experimental evaluations.

Testing was performed with an instrument designed specifically for the purpose of evaluating prophylactic compositions. This device has an adjustable velocity motor-mandrel assembly to which the prophylactic cup is attached, the entirety of which can be automatically moved laterally back and forth by means of a flexible shaft-step motor arrangement. The extent of the lateral movement is precisely controlled by limit switches adjustable to 0.01 inch. The specimen is positioned in a round cup and is held in place by means of a permanent magnet. The specimen

cup is revolved by means of a flexible shaft-variable speed motor assembly, and is positioned on the pan end of a triple-beam balance that enables the precise adjustment of pressure. The prophylactic cup motor assembly is lowered onto the specimen and the height of the specimen cup is adjusted by means of a screw until the force is sufficient to balance the weighted arm of the triple-beam balance. During treatment the prophylactic cup is automatically raised every ten seconds from the specimen surface to simulate slurry replenishment during prophylaxis. For the polishing and abrasion evaluations abrasive slurries were prepared by mixing two parts of a 1% sodium carboxymethyl cellulose solution with one part of abrasive by weight in order to prevent the abrasive from settling.

Polishing evaluations were made with bovine permanent incisors mounted in Wood's metal with the labial surface exposed. The labial surface was leveled by means of a mechanical surface grinder so as to provide a smooth, uniform area for testing that was not into the dentin and was parallel to the base. The teeth selected were of sufficient size to provide a leveled area approximately 1.0 cm in diameter. The mounted tooth specimens were dulled by immersion in 0.2M HCl for thirty seconds, followed by thorough rinsing with distilled water. The specimens were mechanically treated by means of a simulated prophylaxis where the prophylactic cup was rotated under a load of 300 grams at 1000 rpm and oscillated ±0.14 inch from the zero position at a rate of five cycles per minute, and the specimen cup revolved at 30 rpm. This procedure produced a uniformly randomized treatment pattern that ensured reproducibility between replicates. Treatment times were varied from ten seconds to cumulative times of twelve minutes.

The reflectance of the polished tooth surface was determined by means of a reflectometer designed to detect changes in the degree of luster of the enamel surface. This instrument produces a beam of light which, when

reflected from the leveled tooth surface, impinges on a photoelectric cell which in turn activates an X-Y recorder, producing a graphical print-out of the entire leveled tooth surface. The smoother and more highly polished the enamel surface, the smaller is the amount of diffused and absorbed light and, hence, the higher the reflectance reading. The reflectometer was calibrated so that 0 represented total darkness and 100 was set to white carrara glass standards, and the data for the abrasives tested hereinafter are reported on this scale.

The harmfulness of a prophylactic abrasive can be expressed in terms of dentin and enamel abrasion values. Dentin and enamel abrasion values for prophylaxis paste cleaning and polishing agents were determined using bovine teeth. Dentin specimens were prepared by sagittally sectioning bovine permanent incisors through the pulp cavity, carefully trimming off the enamel around the perimeter, and mounting the anterior half in a block of self-curing acrylic with the exposed dentin facing upward. The dentin was ground flush with the acrylic surface using a model trimmer and a coarse wheel. The ground surface was then uniformly smoothed and leveled with a mechanical surface grinder. The dentin specimens were treated for a total of two minutes using the prophylaxis instrument as described previously for enamel polish. Enamel abrasion specimens were prepared by immersing trimmed bovine incisors in dental acrylic with the labial surface facing upward. The blocks were ground, using a model trimmer and coarse grinding wheel, until the enamel was leveled to an area approximately 9 mm in length and was flush with the acrylic. Further smoothing and flattening were accomplished with a mechanical surface grinder, then a very smooth, optically polished surface was produced using 0.3 micron alpha alumina on a horizontal polishing wheel with a silk cover. The enamel specimens were treated for a total of five minutes using the prophylaxis instrument to administer a reciprocating treatment wherein the specimen

was not revolved but maintained stationary while the prophylactic cup made repeated passes over it. All other treatment conditions were the same as described previously.

The amount of dentin or enamel removed by each abrasive under identical treatment conditions was used as an indicator of abrasivity and was quantitatively measured by means of a proficorder, a surface profile measuring device. The proficorder has a diamond stylus which, when tracing the surface of a specimen, produces an electrical signal proportional to the irregularities on the traced surface. These signals are amplified and converted into a graphical form on a strip chart producing a very sensitive, accurate record of the surface microtopography. By superimposing before-and-after treatment traces, the cross-sectional area of abrasion was visualized and was measured by means of a planimeter. From this value the mean abraded depth of each specimen was calculated, and both the dentin and enamel abrasion data are hereinafter reported in this fashion.

The effectiveness of a dental abrasive as a compatible carrier vehicle for fluoride-containing adjuvants was determined by measuring the amounts of available fluoride and tin(II) ions in solution. Percentage availability refers to a comparison of an ionic concentration level with a reference solution of the adjuvant without the carrier vehicle. A percentage ratio of the ionic concentration level detected for each abrasive agent was determined by adding 8.00 grams of abrasive to a 20 ml aliquot of 1000 ppm fluoride solution, mechanically shaking for fifteen minutes, centrifuging until clear, and decanting. The supernatant was analyzed for fluoride with a fluoride electrode and for tin(II) by means of an iodimetric titration. Thus, for example, a combination solution of abrasive (carrier vehicle) and sodium fluoride which analyzed 900 ppm fluoride concentration compared to a reference solution of sodium fluoride at 1000 ppm fluoride exhibits at 90% availability.

Enamel polish data were obtained for a number of prophylaxis abrasives in accordance with this invention. For comparative purposes, data were also obtained for several conventional prophylaxis abrasives as well as a number of magnesium compounds not encompassed by this invention. These data, which are provided in Table 3, show the superior polishing characteristics of the calcined magnesium silicates of this invention.

TABLE 3

ENAMEL POLISH OF CALCINED MAGNESIUM SILICATES AND OTHER ABRASIVES

Polishing Agent	Calcining Temperature	Polish Score (30-Second Treatment) Mean ± Standard Error
Amorphous Hydrous Magnesium Silicate	Uncalcined	64 ± 2
	900°C.	87 + 3
Hectorite	900°C.	99 ± 3
Magnesium Trisilicate	Uncalcined	48 ± 6
	900°C.	89 ± 5
Mica	Uncalcined	54 ± 1
	1100°C.	94 ± 3
Sepiolite	Uncalcined	35 ± 0
	1000°C.	103 ± 3
Chlorite	Uncalcined	38 ± 2
	1200°C.	91 ± 3
Steatite Talc	Uncalcined	35 ± 3
	1200°C.	88 ± 1
Saponite	900°C.	94 ± 2
Cristobalite, SiO_2	--	62 ± 5
Feldspar, $(Na,K)AlSi_3O_8$	--	76 ± 3
Magnesium Carbonate, $MgCO_3$	--	45 ± 3
Magnesium Hydroxide, $Mg(OH)_2$	--	40 ± 3
Magnesium Oxide, MgO	--	62 ± 2
Magnesium Phosphate, $Mg_3(PO_4)_2$	--	49 ± 1
Pumice, Coarse	--	53 ± 1
Pumice, Fine	--	67 ± 3
Pumice, Flour	--	72 ± 2
Quartz, SiO_2	--	65 ± 3
Zirconium Silicate, $ZrSiO_4$	--	74 ± 2

Several of these polishing agents and a number of conventional prophylaxis abrasives were also examined for their rate of enamel polishing ability (i.e. polish versus treatment time). These data, which are reported in Table 4, demonstrate that the materials of this invention produce more rapid rates of polish and higher luster maximums than conventional abrasives used for dental prophylaxis.

TABLE 4

ENAMEL POLISH RATE OF CALCINED MAGNESIUM SILICATES AND OTHER ABRASIVES

Polishing Agent	Calcining Temperature	Polish Score (vs. Treatment Time in Minutes) 1/6	1/2	1	2	4	8	12
Amorphous Hydrous Magnesium Silicate	1000°C.	52	98	106	109	112	112	114
Hectorite	900°C.	81	100	107	113	114	119	120
Magnesium Trisilicate	900°C.	51	90	100	106	111	113	115
Sepiolite	1000°C.	80	103	112	116	116	119	118
Cristobalite	--	39	70	81	90	98	102	105
Feldspar	--	50	76	89	98	105	108	109
Pumice, Coarse	--	32	54	69	84	87	94	94
Pumice, Fine	--	46	64	79	89	96	97	100
Pumice, Flour	--	51	75	86	92	98	103	105
Quartz	--	45	71	90	102	106	107	111
Zirconium Silicate	--	64	85	98	106	111	113	115

Enamel and dentin abrasion data were obtained for several calcined magnesium silicates and conventional prophylaxis abrasives and are reported in Table 5. The data in Table 5 show that the materials of this invention are of low abrasivity to both enamel and dentin.

TABLE 5

ENAMEL AND DENTIN ABRASION OF CALCINED MAGNESIUM SILICATES AND OTHER ABRASIVES

Polishing Agent	Calcining Temperature	Enamel Abrasion Score (micro-in.) Mean± Standard Error	Dentin Abrasion Score (micro-in.) Mean± Standard Error
Amorphous Hydrous Magnesium Silicate	1000°C.	81 ± 16	2031 ± 283
Sepiolite	1000°C.	38 ± 11	715 ± 81
Feldspar	--	90 ± 40	1832 ± 151
Pumice, Coarse	--	967 ± 114	3245 ± 205
Pumice, Fine	--	822 ± 67	3068 ± 478
Pumice, Fluor	--	476 ± 161	2634 ± 327
Zirconium Silicate	--	269 ± 57	1861 ± 55

Fluoride availability data were determined for a number of prophylaxis abrasives in accordance with this invention. For comparison, data were also obtained for conventional prophylaxis abrasives and several magnesium compounds not included in this invention. These data, which are provided in Table 6, demonstrate the high degree of compatibility of the calcined magnesium silicates with three different fluoride-containing adjuvants.

TABLE 6
FLUORIDE COMPATIBILITY OF CALCINED MAGNESIUM SILICATES AND OTHER ABRASIVES

Polishing Agent	Calcining Temperature	Per Cent Availability F (as NaF)	F (as APF)	F (as SnF_2)	Sn++(as SnF_2)
Amorphous Hydrous Magnesium Silicate	Uncalcined	81.3	34.5	67.2	0.2
	1000°C.	99.7	100.3	97.7	96.7
Hectorite	1100°C.	100.4	94.8	99.2	91.8
Magnesium Trisilicate	Unclacined	25.1	5.8	12.4	0.2
	1100°C.	99.3	99.6	98.7	83.8
Mica	Uncalcined	92.0	42.2	60.2	19.7
	1200°C.	100.2	49.6	92.1	85.6
Sepiolite	Uncalcined	43.8	31.3	31.6	0.2
	1100°C.	99.9	84.1	96.9	90.0
Chlorite	Uncalcined	73.0	48.6	65.3	36.4
	1200°C.	100.2	52.4	90.4	77.6
Steatite Talc	Uncalcined	91.6	88.4	90.5	64.8
	1200°C.	99.8	93.9	96.7	86.0
Saponite	1000°C.	99.7	80.6	98.8	92.6
Cristobalite	--	98.3	N.D.	97.2	N.D.
Feldspar	--	99.3	21.3	88.8	59.1
Magnesium Carbonate	--	49.2	12.1	8.5	0.2
Magnesium Hydroxide	--	7.6	25.2	1.7	0.2
Magnesium Oxide	--	0.5	9.0	1.0	3.9
Magnesium Phosphate	--	69.3	17.6	31.5	0.6
Pumice, Coarse	--	98.8	86.7	96.6	71.8
Pumice, Fine	--	97.8	85.9	96.1	70.9
Pumice, Flour	--	89.3	68.5	81.3	56.7
Quartz	--	96.2	N.D.	96.3	N.D.
Zirconium Silicate	--	99.1	N.D.	93.9	N.D.

N.D. -- Not Determined

The foregoing data are supportive of the significant improvement in dental health that may be achieved by utilizing dental prophylactic compositions containing the calcined magnesium silicate cleaning and polishing agents of this invention.

CLAIMS

1. A dental prophylaxis preparation comprising as its principal cleaning and polishing constituent particles of calcined naturally occurring or synthetic magnesium silicate substantially none of which are retained on an 80 mesh screen and essentially all of which pass through a 100 mesh screen, with the median particle size lying in the range of about 10-40 microns.

2. A preparation, as claimed in Claim 1, wherein the calcined magnesium silicate particles are predominantly of the magnesium metasilicate form.

3. A preparation, as claimed in Claim 1, wherein the particles are calcined at a temperature in the range from about 800°C. to about 1200°C.

4. A preparation, as claimed in Claim 1, wherein the naturally occurring magnesium silicate is a member selected from the group consisting of smectite, sepiolite, chlorite, and mica-type minerals.

5. A preparation, as claimed in Claim 1, wherein the magnesium silicate is a member selected from the group consisting of hectorite, montmorillonite, bentonite, saponite, meerschaum, talc, phlogopite, colloidal magnesium silicate, magnesium trisilicate, and amorphous hydrous magnesium silicate.

6. A preparation, as claimed in Claim 1, and further containing a water-soluble, non-toxic anticariogenic adjuvant selected from the group consisting of water-soluble fluoride-containing salts and water-soluble aluminum ion-containing salts.

7. A preparation, as claimed in Claim 1, wherein the calcined magnesium silicate particles are present at a level of about 30-95% by weight of the preparation.

AMENDED CLAIMS

(received by the International Bureau on 18 August 1981 (18.08.81))

1. A dental prophylaxis preparation comprising a carrier and, as its principal cleaning and polishing constituent, particles of a calcined magnesium silicate selected from the group consisting of smectite, sepiolite, chlorite, mica, hectorite, montmorillonite, bentonite, saponite, meerschaum, talc, phlogopite, colloidal magnesium silicate, magnesium trisilicate, and amorphous hydrous magnesium silicate, substantially none of the particles being retained on an 80 mesh screen and essentially all of the particles passing through a 100 mesh screen, with the median particle size lying in the range of about 10-40 microns, the calcined magnesium silicate particles being predominantly of the magnesium metasilicate form.

2. A preparation, as claimed in Claim 1, wherein the particles are calcined at a temperature in the range from about 800°C. to about 1200°C.

3. A preparation, as claimed in Claim 1, and further containing a water-soluble, non-toxic anticariogenic adjuvant selected from the group consisting of water-soluble fluoride-containing salts and water-soluble aluminum ion-containing salts.

4. A preparation, as claimed in Claim 1, wherein the calcined magnesium silicate particles are present at a level of about 30-95% by weight of the preparation.

STATEMENT UNDER ARTICLE 19

The present amendment reduces the number of claims in the application and defines the invention more specifically. The principal cleaning and polishing constituent is now defined as particles of a calcined magnesium silicate from a group of defined materials and which is predominantly of the magnesium metasilicate form.

Although magnesium silicates have been disclosed in prior patents as binders, they have not been proposed for use as cleaning and polishing agents.

INTERNATIONAL SEARCH REPORT

International Application No PCT/US81/00323

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int. Cl.[3] A61K 7/16 A61K 7/18, C 09K 3/14
US Cl. 424/49 424/52, 51/308

II. FIELDS SEARCHED

Minimum Documentation Searched [4]

Classification System	Classification Symbols
US	424/49 to 58 51/308

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [5]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [14]

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No. [18]
A	US, A, 3,105,013, published 24 September 1963 Saul et al	1 to 7
A	US, A, 4,122,163, published 24 October 1978 Muhler et al.	1 to 7
A	US, A, 4,108,979, published 22 August 1978 Muhler et al	1 to 7
A	US, A, 4,108,981 published 22 August Muhler et al	1 to 7
A	US, A, 3,450,813 published 17 June 1969 Muhler et al	1 to 7
A	US, A, 4,064,231 published 20 December 1977 Asakawa et al	1 to 7
A	US, A, 3,855,147 published 17 December 1974 Granquist	1 to 7
A	US, A, 2,059,396 published 03 November 1936 Ripert	1 to 7

* Special categories of cited documents: [15]

"A" document defining the general state of the art
"E" earlier document but published on or after the international filing date
"L" document cited for special reason other than those referred to in the other categories
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but on or after the priority date claimed
"T" later document published on or after the international filing date or priority date and not in conflict with the application, but cited to understand the principle or theory underlying the invention
"X" document of particular relevance

IV. CERTIFICATION

Date of the Actual Completion of the International Search [2]	Date of Mailing of this International Search Report [2]
11 June 1981	22 JUN 1981
International Searching Authority [1]	**Signature of Authorized Officer [10]**
ISA/US	SKRose SK Rose

International Application No. PCT/US81/00323

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No [18]
A	US, A, 2,216,821 published 08 October 1940 Long	1 to 7
A	US, A, 2,384,563 published 11 September 1945 Roseman et al	1 to 7
A	US, A, 1,943,856 published 03 January 1934 Cross	1 to 7
A	DE, B, 1,292,468 published 10 April 1969 C.K. Wolther	1 to 7
A	US, A, 1,049,005 published 31 December 1912 Steinmetz	1 to 7
A	US, A, 1,087,705 published 17 February 1914 Allen	1 to 7
A	US, A, 2,625,514 published 13 January 1953 Kirschenbauer	1 to 7
A	US, A, 2,943,982 published 95 July 1960 Dahlin	1 to 7
A	US, A, 3,966,432 published 29, July 1976 Rayner	1 to 7
A	US, A, 4,038,380 published 26 July 1977 Cordon	1 to 7
A	US, A, 4,169,796 published 02 October 1979 Dahlin	1 to 7
A	US, A, 4,181,633 published 01 January 1980 Colodney et al.	1 to 7